

This Page Is Inserted by IFW Operations  
and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning documents *will not* correct images,  
please do not report the images to the  
Image Problem Mailbox.**

FIG. 1A

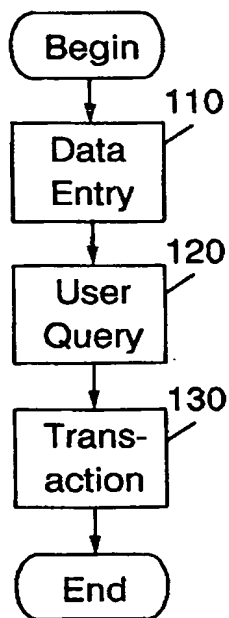


FIG. 1B

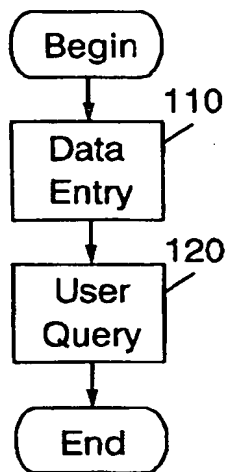


FIG. 1C

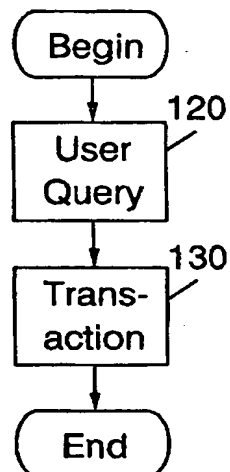


FIG. 1D

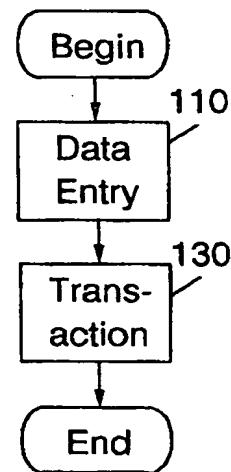


FIG. 1E

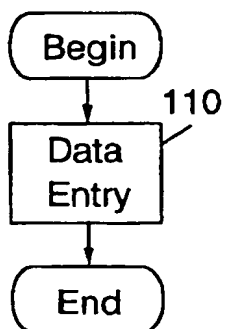


FIG. 1F

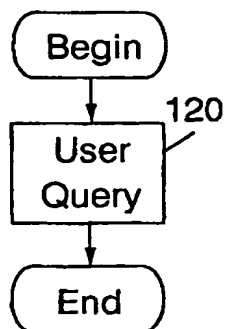


FIG. 1G

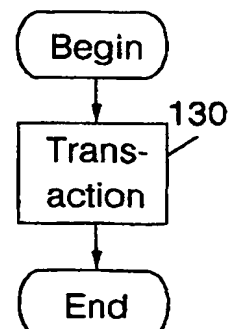


FIG. 2

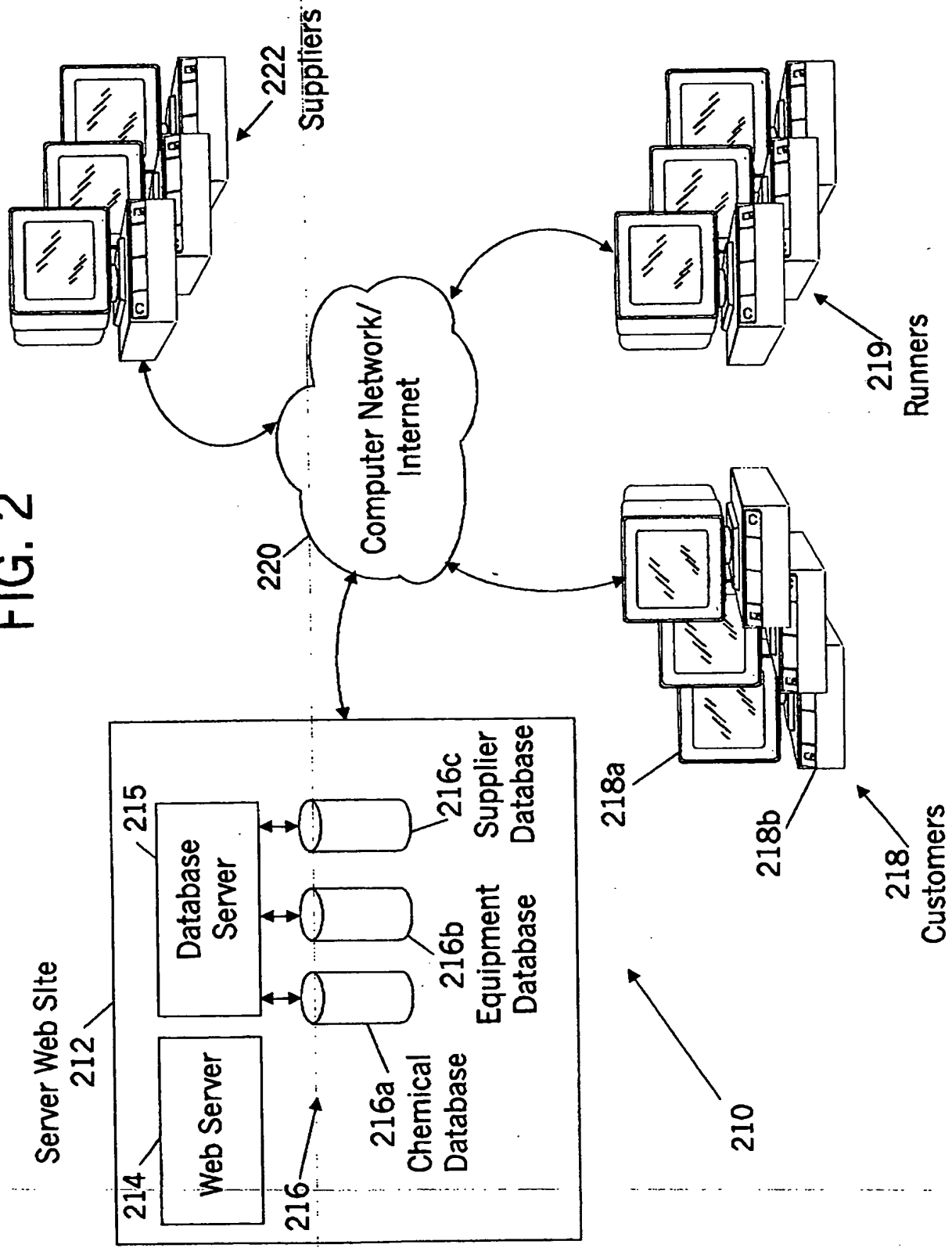
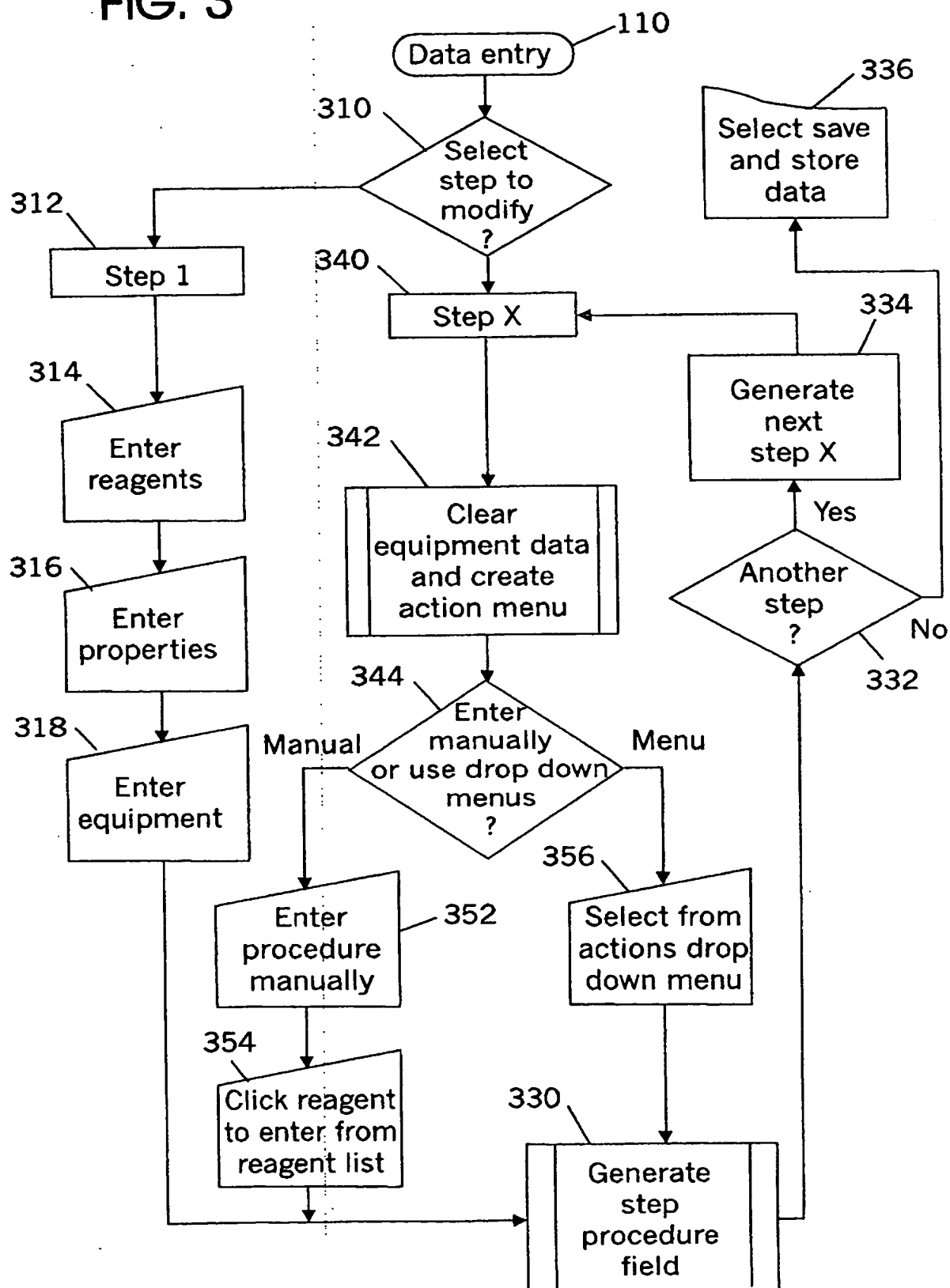


FIG. 3



**FIG. 4**

Synthetix Protocol Manager ver. 0.90b

Name: \_\_\_\_\_ CAS: \_\_\_\_\_ Formula: \_\_\_\_\_ Weight: \_\_\_\_\_

Reagents:   ☐ Enter procedure by hand

**Search Results**

CAS	Chemical Name
71-43-2	Benzene
104-86-9	Benzenemethanamine, 4-chloro-
105-13-5	Benzenemethanol, 4-methoxy-
105-05-5	Benzene, 1,4-diethyl-
118-69-4	Benzene, 1,3-dichloro-2-methyl-
121-14-2	Benzene, 1-methyl-2,4-dinitro-
70-34-8	Benzene, 1-fluoro-2,4-dinitro-
93-05-0	1,4-Benzenediamine, N,N-diethyl-
98-10-2	Benzenesulfonamide
104-51-8	Benzene, butyl-
827-52-1	Benzene, cyclohexyl-
14321-27-8	Benzenemethanamine, N-ethyl-

**Procedure**

Step 2

Qty

Qty

Qty

Qty

Qty

Qty

Qty

Qty

Qty

Qty

FIG. 5

6222750

006310" 6232/60

Synthematix Protocol Manager ver. 0.90b

Name:

Reagents

CAS	Name	Weight	Formula	Density	BP	FP	MP	Vapor Pressure	Comments	Ballston	Other Names
101-01-1											

Procedure

Previous:

Save

Cancel

Exit

New

Save

Update

Add To Procedure

FIG. 6

Synthematix Protocol Manager ver. 0.90b

Name:  CAS:  Formula:  Weight:

Reagents:   ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	Action
1	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
2	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
3	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
4	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
5	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
6	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
7	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
8	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
9	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
10	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>

Qualifier:

Action:

Reagent:

Time:

Procedure

Step 2

Yield:  Density:

BP:  MP:

MP:  Vapor Pr:

Ballstien:  Other Names:

FIG. 7





TOP SECRET 6232/650

Synthematix Protocol Manager ver. 0.90b

Name: CAS Formula Weight

Reagents:   ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	Action
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

Search Results

- Class stir rod
- Overhead stirrer
- Teflon stir paddle

Procedure

Qty		Qty	
	stir		

FIG. 9

Synthetix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene

	CAS	Name	Weight	Amount (g)
1	R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50
2	107-11-9	2-Propen-1-amine	57.09	100
3	64-17-5	Ethanol	46.07	100
4	71-43-2	Benzene	78.11	3146.4
5				
6				
7				
8				
9				
10				

Starting Flask: Round bottom 3-neck flask ☒

Equipped with:

	<input type="checkbox"/>
	<input type="checkbox"/>
	<input type="checkbox"/>
	<input type="checkbox"/>
	<input type="checkbox"/>
	<input type="checkbox"/>
	<input type="checkbox"/>
	<input type="checkbox"/>
	<input type="checkbox"/>
	<input type="checkbox"/>

Procedure

Step 1

Into a round bottom 3-neck flask was added <GN0> grams (<MN0> mol) of 5-amino-4,6-dichloropyrimidine

FIG. 10

Synthematix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene  ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	Action
1	R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50	
2	107-11-9	2-Propen-1-amine	57.09	100	
3	64-17-5	Ethanol	46.07	100	
4	71-43-2	Benzene	78.11	3146.4	
5					
6					
7					
8					
9					
10					

Qualifier:

Action:

Time:

Procedure

Step: 7

FIG. 11

Synthematix Protocol Manager ver. 0.90q

File Apparatus Atmosphere Operations

Name: 6-(allylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene  ☐ Enter procedure by hand

CAS	Name	Weight	Amount (g)
R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50
107-11-9	2-Propen-1-amine	57.09	100
64-17-5	Ethanol	46.07	100
71-43-2	Benzene	78.11	3146.4

Action:   was   added   Reagent 2

Procedure

Step 2

To the flask was added <GN1> grams (<MN1> mol) of 2-Propen-1-amine and <GN2> grams (<MN2> mol) of Ethanol

FIG. 12



**Synthematix Protocol Manager ver. 0.90a**

File Apparatus Atmosphere Operations

Name: **6-(allylamino)-5-amino-4-chloropyrimidine** CAS: **R00-02-3** Formula: **C7H9N4Cl** Weight: **184.63**

Reagents: **benzene**  ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	
1	R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50	Action: <input type="text"/>
2	107-11-9	2-Propen-1-amine	57.09	100	Qualifier: <input type="text"/>
3	64-17-5	Ethanol	46.07	100	Action: <input type="text"/>
4	71-43-2	Benzene	78.11	3146.4	Reagent: <input type="text"/>
5					Time: <input type="text"/>
6					<input type="button" value="Reset"/> <input type="button" value="Add To Procedure"/>
7					
8					
9					
10					

**Procedure**

Step 2

To the flask was added <GN1> grams (<MN1> mol) of 2-Propen-1-amine and <GN2> grams (<MN2> mol) of Ethanol

FIG. 13

6333/50

Synthematix Protocol Manager ver. 0.90b

Name: \_\_\_\_\_ CAS: \_\_\_\_\_ Formula: \_\_\_\_\_ Weight: \_\_\_\_\_

Reagents:   ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	Action
1					
2					
3					
4					
5					
6					
7					
8					
9					
0					

Qualifier:

Action:

Reagent:

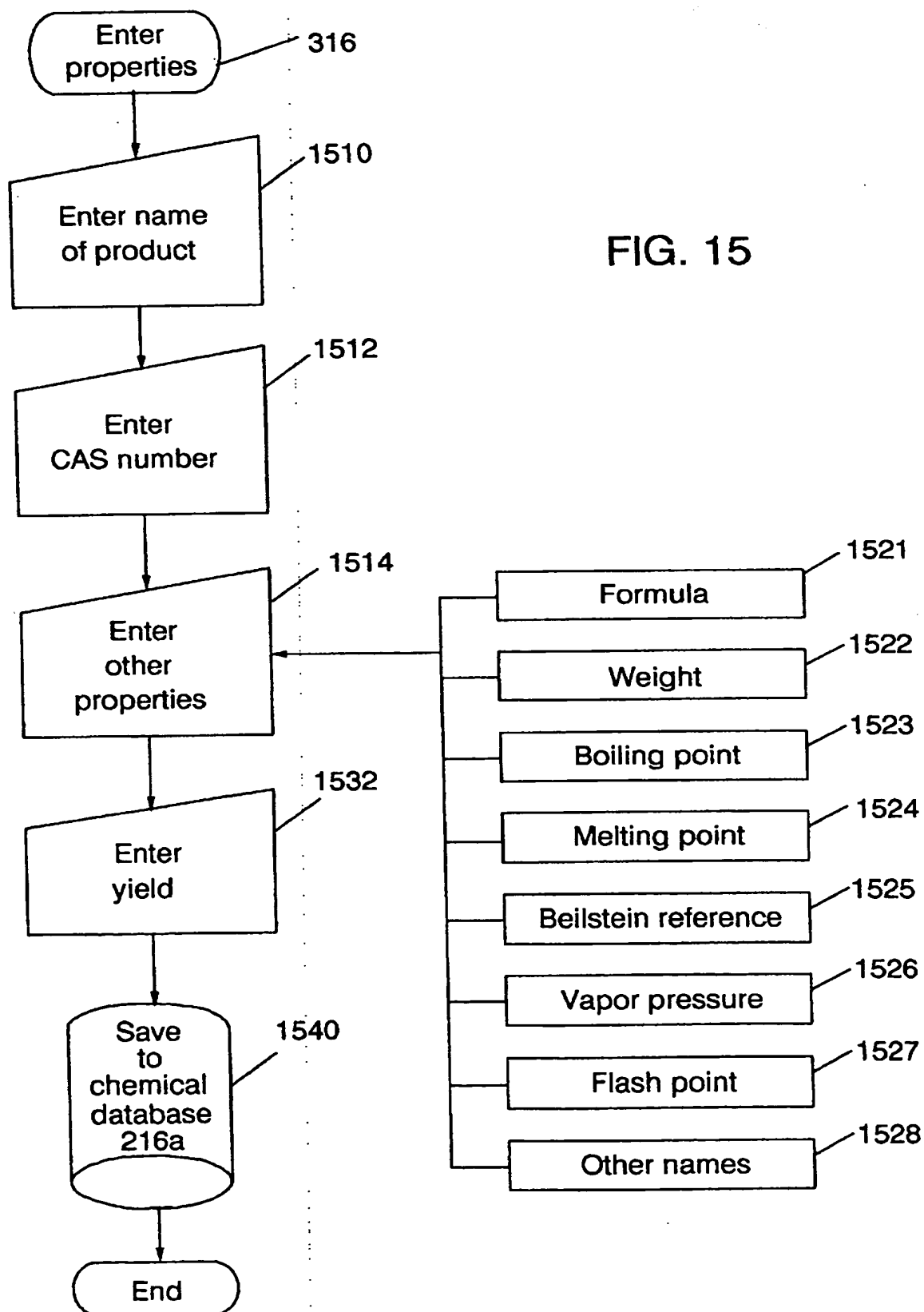
Time:

Procedure

Step 2

Reaction Type/Keywords (Separated by semicolon)

FIG. 14





File Apparatus Atmosphere Operations

Name: 6-(ethylamino)-5-amino-4-chloropyrimidine CAS: R00-02-3 Formula: C7H9N4Cl Weight: 184.63

Reagents: benzene  ☐ Enter procedure by hand

CAS	Name	Weight	Amount (g)	Action
R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50	<input type="button" value="Add"/>
107-11-9	2-Propen-1-amine	57.09	100	<input type="button" value="Add"/>

6-

Author: Thomas et al. Journal: acid chemistry  
 Year: 1968 Vol: 1  
 Page: 22

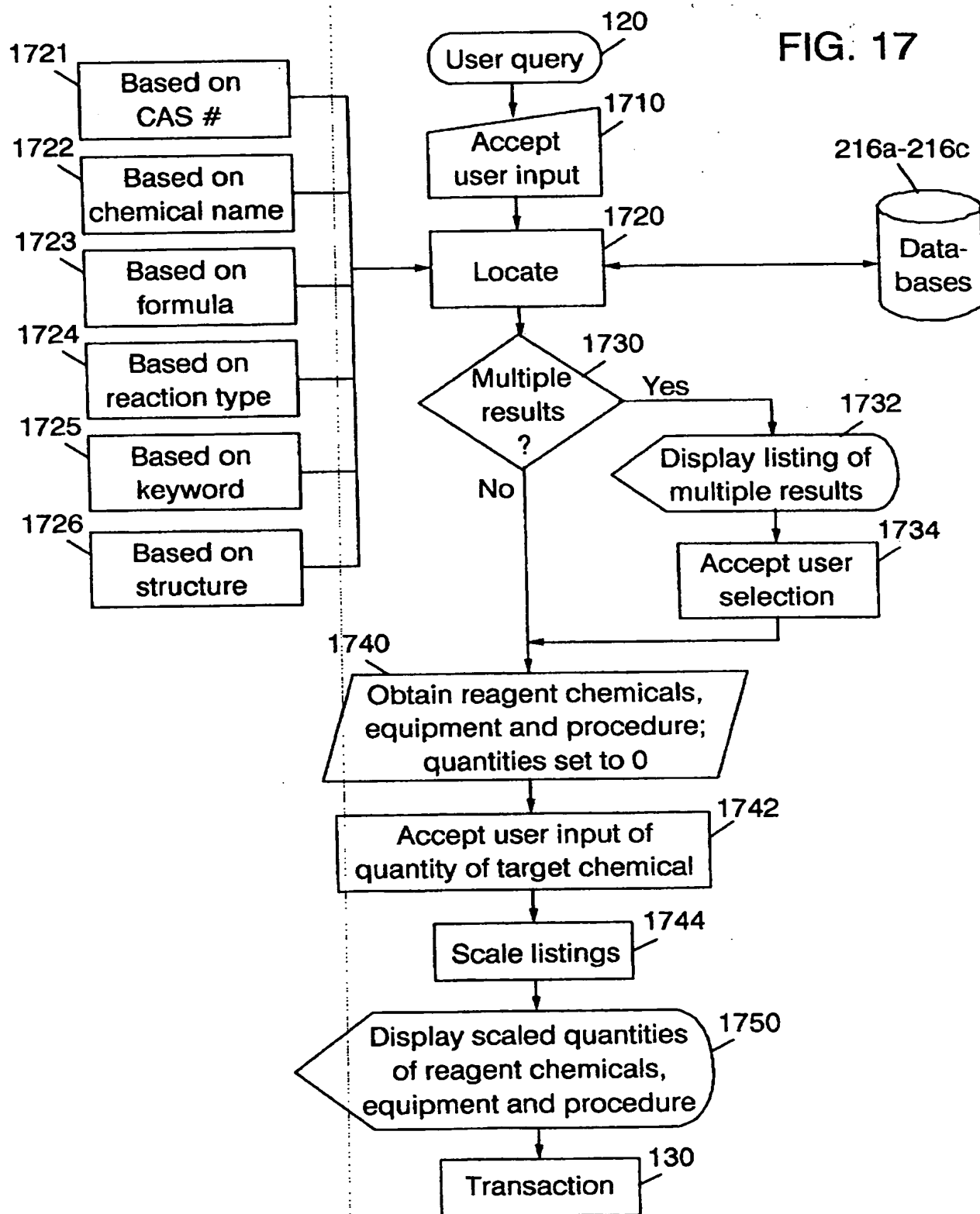
Procedure

Step 6

The extracts are then evaporated to dryness and can be recrystallized from petroleum ether to give a pure product

FIG. 16

FIG. 17




Synthematrix: Powered by Scientists - Netscape

Back Forward Reload Home Search Netscape Print Security Shop Stop

Bookmarks Location: [http://63.119.160.203/secure/search\\_chembase.html](http://63.119.160.203/secure/search_chembase.html) What's Related

**CLICK ME.**

 **SYNTHEMATIX**  
Powered by Scientists™

Search | Submit News | Submit Publication | Advertising | Privacy Policy | User Preferences | About | Feedback | Help Wanted

<p><b>Search for:</b></p> <p><b>Chemical Procedures</b> Access our database of over 10,000 chemical procedures</p> <p><u>Equipment</u> Shop for lab equipment</p> <p><u>Raw Materials</u></p> <p><u>Glassware</u> Stock and custom glassware</p> <p><u>Publications</u> Access scientific papers published online with Synthematrix</p> <p><u>Advanced</u> Enter an advanced site-wide search query</p> <p><b>Submit:</b></p> <p><u>Publication</u> Share your research with the world wide science community!</p> <p><u>News</u> Submit URL for the latest news stories of interest to the Synthematrix community.</p>	<p><b>Search Chemical Procedure Database</b></p> <p><b>If you know what you need</b></p> <p>Enter the compound CAS # (Example: 514-10-3) or the chemical formula (Example: C5H2) or the compound name (Example: methanol)</p> <p><input type="text" value="bromo"/></p> <p><input type="button" value="Locate"/></p> <p><b>If you don't know exactly</b></p> <p>Enter the type of reaction you are browsing here. (Example: reduction, addition, etc)</p> <p><input type="text"/></p> <p><input type="button" value="Locate Reaction Type"/></p>	<p><b>User Login</b></p> <p>login: <input type="text"/></p> <p>password: <input type="password"/></p> <p><input type="button" value="User Login"/></p> <p>Login to your account to use personalized functions of this site. If you don't have an account <a href="#">create one.</a></p>
---	--	--

**Synthematrix: Powered by Scientists**

Document: Done


FIG. 18

Synthematix: Search Results - Netscape

File Edit View Go Communicator Help

Back Forward Reload Home Search Netscape Print Security Shop Stop

Bookmarks Location http://63.119.160.203/servlets/QueryProject?name=bromo What's Related

 **SYNTHEMATIX**  
Powered by Scientists™

**Chemical Procedures**

**Search for:**  
[Chemical Procedures](#)  
 Access our database of over 10,000 chemical procedures  
[Equipment](#)  
 Shop for lab equipment  
[Raw Materials](#)  
[Glassware](#)  
 Stock and custom glassware  
[Publications](#)  
 Access scientific papers published online with Synthematix  
[Advanced](#)  
 Enter an advanced site-wide search query

**Synthematix: Search Results**

Number of matches: 7

[alpha-bromo-p-toluene boronic acid](#)  
[n-Butyl-2-bromopropionate](#)  
[\(6-Bromo-2,2-diphenylbenzo\[1,3\]dioxol-5-yl\)methanol](#)  
[\(6-Bromo-2,2-dimethylbenzo\[1,3\]dioxol-5-yl\)methanol](#)  
[6-bromo-2,2-diphenylbenzo\[1,3\]dioxole-5-carbaldehyde](#)  
[6-bromo-3,4-\[\(diphenylmethylene\)dioxy\]-benzaldehyde ethanediyl acetal](#)  
[1-Benzhydryl-3-bromoazetidine](#)

*Synthematix: Powered by Scientists*

[home](#) | [about](#) | [chemical procedures](#) | [equipment](#) | [glassware](#) | [raw materials](#) | [privacy policy](#) | [submit publication](#) | [submit news](#) | [advertising](#)

Copyright 2000 Synthematix. All trademarks and copyrights on this page are owned by their respective companies. Comments are owned by the Poster.

Document Done

FIG. 19

T06220"6222/650

2010

Synthetic Compound Procedure - Microsoft Internet Explorer

Address: <http://63.119.160.203/servlets/ShowChembase?id=16>

2-Butyl-2-bromopropionate (209.08) C7H13BrO3

2-Bromopropionic acid  
 $C_3H_5BrO_2$   
 Exact Mass: 151.98  
 Mol. Wt: 152.98  
 C: 23.55, H: 3.27, Br: 52.23, O: 20.92

1-Butanol  
 $C_4H_{10}O$   
 Exact Mass: 74.07  
 Mol. Wt: 74.12  
 C: 64.82, H: 13.50, O: 21.59

n-Butyl-2-bromopropionate  
 $C_7H_{13}BrO_2$   
 Exact Mass: 208.01  
 Mol. Wt: 209.08  
 C: 40.21, H: 6.27, Br: 38.22, O: 15.30

2060

2020

Enter Mol Scale:  mol

Chemicals needed

Chemical	Formula	Weight	Equivalent	Moles Needed	Grams Needed
2-Bromopropionic acid		152.98	1	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>
n-butanol		74.12	1	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>
Hexane		86.18	1	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>
Dowex 50W4-200		3.68		<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>
Water		18	1	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>

2030

Equipment needed

Qty	Equipment
1	Round bottom 3-neck flask
1	Overhead stirrer
1	Dean Stark Trap
1	Condenser, Allihn, Drip tip
1	Heating mantle
1	Recirculating Chiller
1	Heavy duty distillation head
1	Fraction Cutter
4	500 ml round bottom flask
2	Varisc
3	Tubing (N.)

2040

Procedure

1. Into a 3 Neck Flask equipped with an overhead stirrer, Dean stark collector, condenser, and heating mantle was placed  grams ( mol) of 2-Bromopropionic acid.
2. To the 2-Bromopropionic acid was added  grams ( mol) of Butanol.
3. To the 2-Bromopropionic acid/butanol mix was added  grams ( mol) of Hexane.
4. To the flask is added  grams of Dowex resin to facilitate the esterification. The resin is previously dried and has an activity of 3.68 grams/mol.
5. Two variscs are attached to the heating mantle and a setting of 50% power is used for heating to rapid reflux.
6. The reaction is monitored for water generation. After collecting  grams ( mol) of water the reaction is complete.
7. Hexane is then removed by continuous draining of the dean stark collector.
8. The reaction is then cooled to room temperature and filtered through a fritted glass funnel to remove the Dowex resin.
9. The crude n-butyl-2-bromopropionate is then purified via vacuum distillation. (The vacuum distillation procedure is below)

Vacuum distillation

- The product is placed into a 5L round bottom and connected to the pilot scale distillation head equipped with a vigorous column. The material is heated in a 5L mantle set to 45-50% power with a varisc.
- The product is collected at a head temp of 40-45°C. The material rapidly condenses once the apparatus is heated up. Total time for distillation is approx. 5 hours.

2050

References

FIG. 20

2010  
2060  
2020  
2030  
2040  
2050

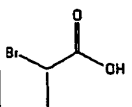
Synthetic Compound Procedure - Microsoft Internet Explorer

Back Forward Stop Refresh Home Search Favorites History Mail Print Edit Discuss Real.com


Address: [7id=16&mol=12&MN0=1835.76&MN1=12&GN1=889.44&MN2=12&GN2=1034.16&MN3=12&GN3=44.16&MN4=12&GN4=216](#)

Links: [crn](#) [Science resource](#) [Internet Start](#) [Windows Update](#) [RealPlayer](#) [HotBot](#) [To Yahoo!](#)

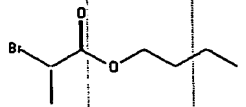
**n-Butyl 2-bromopropionate (209.08)** **C7H13BrO3**



2-Bromopropionic Acid  
C<sub>3</sub>H<sub>5</sub>BrO<sub>2</sub>  
Exact Mass: 151.95  
Mol. Wt.: 152.98  
C, 23.55; H, 3.2%;  
Br, 52.23, O, 20.92



1-Butanol  
C<sub>4</sub>H<sub>10</sub>O  
Exact Mass: 74.07  
Mol. Wt.: 74.12  
C, 64.82; H, 13.50, O, 21.59



n-Butyl 2-bromopropionate  
C<sub>7</sub>H<sub>13</sub>BrO<sub>2</sub>  
Exact Mass: 208.01  
Mol. Wt.: 209.08  
C, 40.21; H, 6.27; Br, 38.22, O, 15.30

+ H<sub>2</sub>O

Enter Mol Scale:  mol

**Chemicals needed**

Chemical	Formula Weight	Equivalents	Moles Needed	Grams Needed
2-Bromopropionic acid	152.98	1	<input type="text" value="12.0000"/>	<input type="text" value="1835.7600"/>
n-butanol	74.12	1	<input type="text" value="12.0000"/>	<input type="text" value="889.4401"/>
Hexane	86.18	1	<input type="text" value="12.0000"/>	<input type="text" value="1034.1600"/>
Dowex 50W4-200	3.68	1	<input type="text" value="12.0000"/>	<input type="text" value="44.1600"/>
Water	18	1	<input type="text" value="12.0000"/>	<input type="text" value="216.0000"/>

**Equipment needed**

Qty	Equipment
1	Round bottom 3-neck flask
1	Overhead stirrer
1	Dean Stark Trap
1	Condenser, Allihn, Drip tip
1	Heating mantle
1	Recirculating Chiller
1	Heavy duty distillation head
1	Fraction Cutter
4	100 ml round bottom flask
2	Varisc
3	Tubing (ft.)

**Procedure**

- Into a 3 Neck Flask equipped with an overhead stirrer, Dean stark collector, condensor, and heating mantle was placed  grams ( mol) of 2-Bromopropionic acid.
- To the 2-Bromopropionic acid was added  grams ( mol) of Butanol.
- To the 2-Bromopropionic acid/butanol mix was added  grams ( mol) of Hexane.
- To the flask is added  grams of Dowex resin to facilitate the esterification. The resin is previously dried and has an activity of 3.68 grams/mol.
- Two variscs are attached to the heating mantle and a setting of 50% power is used for heating to rapid reflux.
- The reaction is monitored for water generation. After collecting  grams ( mol) of water the reaction is complete.
- Hexane is then removed by continuous draining of the dean stark collector.
- The reaction is then cooled to room temperature and filtered through a fritted glass funnel to remove the Dowex resin.
- The crude n-butyl-2-bromopropionate is then purified via vacuum distillation. (The vacuum distillation procedure is below)

**Vacuum distillation**

- The product is placed into a 5L round bottom and connected to the pilot scale distillation head equipped with a vigreux column. The material is heated in a 5L mantle set to 45-50% power with a varisc.
- The product is collected at a head temp of 40-45°C. The material rapidly condenses once the apparatus is heated up. Total time for distillation is approx. 5 hours.

**References**

FIG. 21

FIG. 22

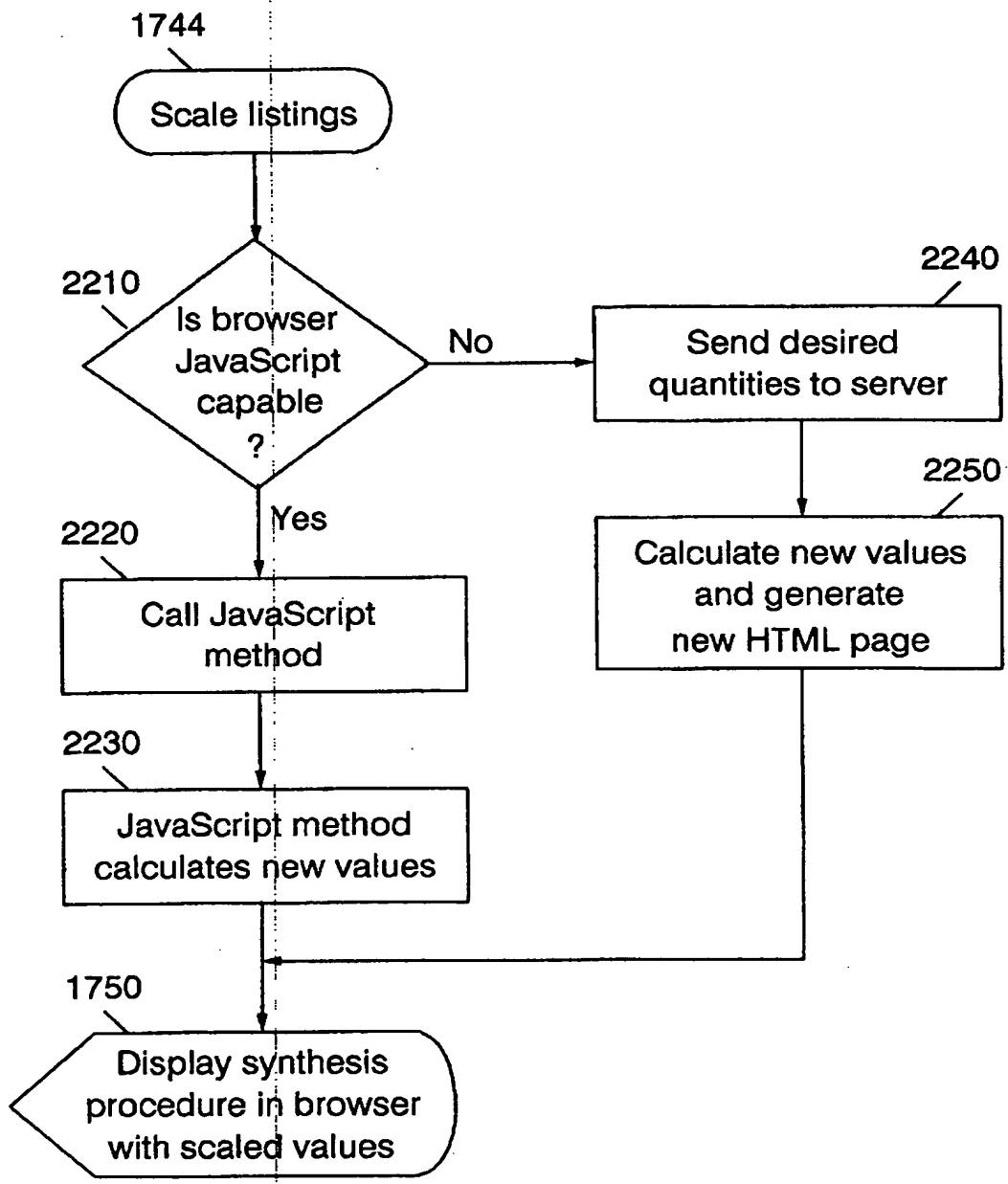


FIG. 22

FIG. 23

